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54 **Stabilized solid compositions.**

57 A solid composition containing a monosaccharide or/and a disaccharide and an amino acid is stabilized by incorporating therein at least 40%, based on said amino acid, of a polysaccharide with a water content of not more than 3%.

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Stabilized Solid Compositions

This invention relates to the stabilization of solid compositions. More particularly, this invention relates to the stabilization of a solid composition containing a monosaccharide or/and a disaccharide and at least one amino acid.

A solid composition containing monosaccharide or/and disaccharide and amino acid, particularly a powdery composition containing them, is known to undergo browning and caking, due to the interaction of saccharide with amino acid, and in the food and pharmaceutical industries, for instance, there has been a long-standing need to establish a technology for preventing and arresting such browning and caking phenomena.

Under the technical circumstances the present inventors conducted an intensive research and unexpectedly found that both the browning and caking phenomena could be substantially prevented by incorporating in such a solid composition at least 40% (weight/weight) [hereinafter all the percent is expressed as weight/weight percent] of a polysaccharide with a water content of not more than 3%, relative to said amino acid. The finding was followed by further studies which have resulted in the accomplishment of this invention.

Thus, this invention provides a method of stabilizing a solid composition characterized by incorporating in a solid composition containing a monosaccharide or/and a disaccharide and an amino acid at least 40 %, based on said

amino acid, of a polysaccharide with a water content of not more than 3 %, and also to a solid composition thus stabilized.

5 The stabilizing method of this invention is applicable to any solid composition containing optional kinds of monosaccharide or/and disaccharide and amino acid that would otherwise undergo browning or/and caking. Said monosaccharide may be any of tetroses, pentoses and hexoses, and is exemplified by erythrose, threose, ribose, xylose,
10 arabinose, glucose, fructose, mannose, galactose, etc. As examples of said disaccharide may be mentioned sucrose, lactose, maltose, etc. Particularly preferred species of these saccharides are glucose, fructose, xylose, galactose, sucrose, lactose, maltose, etc. Only one member of these
15 monosaccharides and disaccharides may be present in said solid composition or two or more members of them may be present. (Hereinafter, these monosaccharides or/and disaccharides will be referred to briefly as "sugar".)

The amino acid may be any of aliphatic amino acids
20 such as monoaminomonocarboxylic acids (glycine, alanine, valine, leucine, isoleucine, etc.), hydroxyamino acids (serine, threonine, etc.), sulfur-containing amino acids (cysteine, cystine, methionine, etc.), monoaminodicarboxylic acids (aspartic acid, glutamic acid, etc.), diaminomono-
25 carboxylic acids (lysine, arginine, etc.), amino acids containing an aromatic nucleus (tyrosine, phenylalanine, etc.), and amino acids containing a heterocyclic ring (histidine, tryptophan, proline, hydroxyproline, etc.). These amino acids may be in the form of salts with physi-
30 ologically acceptable bases or acids. Said bases include inorganic bases such as alkali metals (sodium, potassium, etc.), and alkaline earth metals (calcium, barium, etc.), and said acids include mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid, etc. and organic
35 acids such as acetic acid, lactic acid, fumaric acid, tartaric acid, citric acid, gluconodeltalactone, gluconic

acid, malic acid, etc. Among these amino acids, glycine, alanine, methionine, aspartic acid and its sodium salt, and glutamic acid and its sodium salt are particularly suitable. Either one member or more members than one such
5 amino acid may be present in said solid composition.

The polysaccharide is preferably composed of at least 4 monosaccharide units, such as starch, cellulose, dextran, pullulane, agar, pectin, konniyaku mannan, starch phosphate ester sodium, arabinogalactan, dextrin, cyclodextrin, etc.
10 Particularly preferred are those consisting of glucose units and, for example, starch, pullulane, dextrin, cyclodextrin, etc. are advantageous. It is especially advantageous to use dextrin.

In accordance with this invention, the above-mentioned
15 polysaccharide, in a state containing not more than 3 % of water and in a proportion of at least 40 % relative to the amino acid, is incorporated in said solid composition containing the sugar and amino acid, whereby said browning and caking are effectively prevented. Particularly, the
20 browning and caking can be prevented very remarkably by adding the polysaccharide in a state containing not more than 2 % and, especially, not more than 1 % of water. Such stabilizing effect cannot be accomplished with the polysaccharide whose water content is in excess of 3 %
25 which is the usual level.

Relative to the total weight of amino acids in the composition, the polysaccharide is desirably incorporated in a proportion of about 40 to 800 %, preferably 60 to 700 % and for still better results, 90 to 500 %.

30 To incorporate the polysaccharide with a water content of not more than 3 %, preferably not more than 2 % and more desirably not more than 1 % in said solid composition, the water content of the polysaccharide may be previously adjusted to the defined level before addition
35 to the solid composition or the polysaccharide with an usual water content is added to the solid composition and

the mixture subsequently dehydrated to a water level of not more than 3 %, preferably not more than 2 % or more desirably not less than 1 %. In incorporating the polysaccharide, it is advantageous to admix the polysaccharide with the sugar, amino acid and other components to be incorporated in the desired solid composition, all in powdery form. It is generally advisable to prepare each component in suitable grain size before admixing. It is especially advantageous to evenly compound the components after passing them through a mesh of 42 mesh size (mesh size referred to in "Japan Industrial Standard Z 8801-1976", the same applies hereinafter) or less, especially a mesh of 60 mesh size. When particles in excess of 42 mesh are employed, it is desirable to prepare them to a uniform size. The composition after addition of the polysaccharide may be granulated or tableted by the conventional procedure, if desired. The resulting preparation is substantially free from both the borwning and caking tendencies.

In the practice of this invention, a more pronounced stabilizing effect can be realized by maintaining the polysaccharide-containing composition under conditions that will substantially prevent ingress of water into the composition. Particularly, under high temperature, high humidity conditions, the composition is desirably encased or otherwise sealed against the external environment as soon as possible after compounding. For this purpose, it is advantageous to package the composition gas-tight using a gas-impermeable or sparingly gas-permeable packaging material such as a plastic sheet, a glass container, polyvinyl chloride, polypropylene, cellophane or polyvinylidene chloride copolymer film, or a suitable laminate material, to name but a few. In case a relatively permeable material such as polyethylene is used as said packaging material, it is desirable to use one having a comparatively large thickness.

The preparation and packaging of the solid composition

according to this invention are preferably conducted in an environment at a relative humidity of not more than 70 %, preferably not more than 60 %, and a temperature of not higher than 30°C, preferably not higher than 25°C.

5 The stable composition according to this invention may contain optional components in addition to the sugar, amino acid and polysaccharide according to the intended use. Such additional components may for example be a variety of flavorants (e.g. sweeteners such as saccharin, 10 saccharin sodium, glycyrrhizin, stevioside, etc.), medicaments, salts, preservatives, fungicides, volume builders, etc. The reaction between sugar and amino acid is promoted by the presence of electrolytes such as salts, especially acetates (e.g. sodium acetate, potassium acetate, 15 etc.), carbonates (e.g. sodium carbonate, magnesium carbonate, calcium carbonate, etc.) or hydrogen carbonates (e.g. sodium hydrogen carbonate, etc.) but this invention is effective enough to stabilize even a composition containing about 1 to 20 %, and particularly 3 to 15 %, of acetate, 20 carbonate or/and hydrogen carbonate.

 This invention is applicable to a broad range of sugar-and amino acid-containing solid compositions and especially to sugar-and amino acid-containing powdery compositions such as powdery foods, condiments, juices, 25 drugs for humans, veterinary drugs, feed additives, feeds and so on and as it attains a significant inhibition of browning and caking of such compositions during production, storage, transit and distribution, it is of great commercial value. Particularly, this invention provides a very re- 30 markable stabilization of a powdery electrolyte modifier for animal use which contains glucose, glycine and electrolytes such as sodium chloride, monopotassium phosphate, sodium citrate, magnesium sulfate and sodium hydrogen carbonate.

35 The effectiveness of this invention will hereinafter be described in detail by way of experimental and working

examples, it being, however, to be understood that the invention is by no means limited thereto. It should also be noted that the production and packaging of solid compositions in the following experimental and working examples were conducted at a relative humidity of 50 to 60 % and a temperature of 13 to 20°C unless otherwise specified.

Throughout the present specification as well as in claims, the abbreviations "g", "l", "°C" and "R.H." respectively refer to "grams(s)", "liter(s)", "degree(s) centigrade" and "relative humidity", and percent is weight/weight percent unless otherwise specified.

Experimental Example 1

(1) Test method

Fifty parts (by weight, the same applies hereinafter) of anhydrous glucose, 50 parts of glycine and, based on the glycine, 30, 40, 60, 80 or 100% of dry dextrin (water content 1.5%), all components having been passed through a mesh of 60 mesh size, were evenly compounded and packaged gas-tight in a laminate bag. Each of the bags was stored in an incubator maintained at 40°C and 80 % R.H. and the content was compared with a control preparation which did not contain dextrin as to the degrees of browning and caking.

(2) Results

The results of the above experiment are shown in Table 1. The sample containing 40 % of dextrin relative to glycine was superior to control, and the preparations containing 60, 80 and 100 % of dextrin were very stable without showing any appreciable browning or caking even after 25 days of storage. In the table, - denotes no browning, ± faint browning, + through ++ various degrees of browning. (The like legends hereinafter mean the like effects.)

Table 1

Composition (parts)			Amount of dextrin relative to glycine (%)	Stability as the lapse of storage days							
Anhydrous glucose	Glycine	Dextrin		3	5	10	13	20	25	30	40
50	50	0	0	+	+	+	+	+	+	+	+
50	50	15	30	-	-	+	+	+	+	+	+
50	50	20	40	-	-	-	+	+	+	+	+
50	50	30	60	-	-	-	-	-	-	-	-
50	50	40	80	-	-	-	-	-	-	-	-
50	50	50	100	-	-	-	-	-	-	-	-

Experimental Example 2

(1) Test method

Forty parts of anhydrous glucose were compounded with 5, 10 or 15 parts of glycine and, based on the glycine, 40, 60, 80 or 100 % of dry dextrin (water content 2%), all components having been passed through a mesh of 60 mesh size, and each of the resulting compositions was tested by the same procedure as Experimental Example 1. For the purpose of promoting browning, 10 parts of sodium carbonate (anhydrous) was added to the composition.

(2) Results

The results of the above experiment are shown in Table 2. The samples containing 40, 60, 80 or 100 % of dextrin relative to glycine showed excellent stability despite the quantitative variation of glycine relative to glucose and the presence of sodium carbonate.

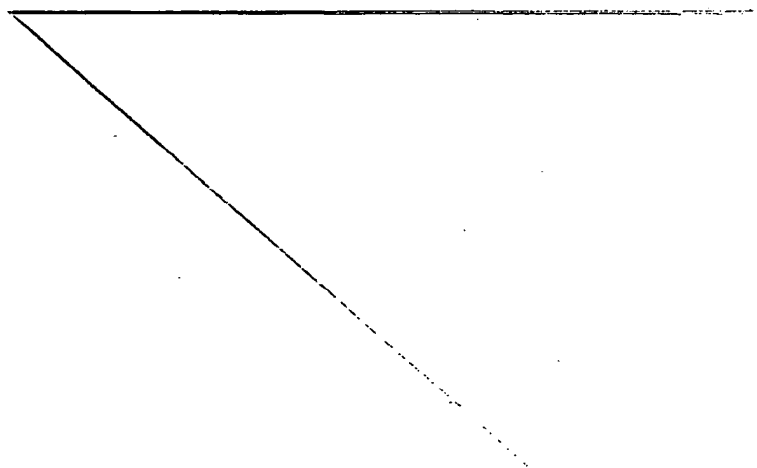


Table 2

[illegible]

Experimental Example 3

(1) Test method

Forty parts of anhydrous glucose, 15 parts of glycine and 10 parts of sodium carbonate (anhydrous) and dextrin having a varying water content of 1%, 2%, 3%, or 4%, all component having been passed through a mesh of 60 mesh size, were admixed and the stability of the resulting mixtures was determined by the same procedure as Experimental Example 1. Dextrin was incorporated in a proportion of 100 % relative to glycine.

(2) Results

The results of the above experiment are shown in Table 3. While the sample containing 4 % of water was not much different from the control (without dextrin), the samples containing 3 % or less, particularly one containing 1 % of water, exhibited excellent stability.

Classification	Composition (parts)				Water content of dextrin (%)	Stability as the lapse of storage days							
	Anhydrous glucose	Glycine	Sodium carbonate	Dextrin		4	5	6	10	15	20	25	30
Control	40	15	10	--	--	+	+	+	+	+			
Control	40	15	10	15	4	-	+	+	+	+		+	
This invention	40	15	10	15	3	-	-	-	+	+	+		+
This invention	40	15	10	15	2	-	-	-	-	-	-	-	+
This invention	40	15	10	15	1	-	-	-	-	-	-	-	-

Experimental Example 4

(1) Test method

Thirty parts of fructose, 30 parts of L-aspartic acid and, based on the aspartic acid, 20, 30, 40, 50 or 100 % of purified and sterilized dry corn starch (water content 1 %) were respectively passed through a mesh of 60 mesh size and admixed. The stability of these compositions was determined by the same procedure as Experimental Example 1. For the purpose of promoting discoloration, 10 parts of sodium acetate (anhydrous) was added to each composition.

(2) Results

The results of the above experiment are shown in Table 4. The sample containing 40 % of purified and sterilized dry corn starch relative to L-aspartic acid was superior to control and the sample containing 50 % of corn starch was still better. The sample containing 100 % exhibited excellent stability without showing any discoloration even after 30 days of storage.

Table 4

Composition (parts)				Amount of the corn starch relative to L-aspartic acid(%)	Stability as the lapse of storage days						
Fructose	L-aspartic acid	Sodium acetate	Purified and sterilized dry corn starch		2	5	7	10	15	20	30
30	30	10	0	0	+	+	+	+	+		
30	30	10	6	20	-	-	+	+	+	+	
30	30	10	9	30	-	-	-	+	+	+	+
30	30	10	12	40	-	-	-	-	-	+	+
30	30	10	15	50	-	-	-	-	-	-	+
30	30	10	30	100	-	-	-	-	-	-	-

Example 1

Using as a control a powdery condiment prepared by admixing 46% of sodium chloride, 26% of anhydrous glucose, 1% of disodium succinate, 0.05% of 5'-ribonucleotide sodium and 26.95% of sodium L-glutamate, each of the components having been previously passed through a mesh of 60 mesh size, a stability test was conducted by the same procedure as Experimental Example 1 on a composition prepared by adding 20 parts (96.2% based on the sodium L-glutamate) of dextrin [Pinedex^R, Matsutani Chemical Co., Ltd. Itami, Japan], which had been thermally dehydrated to a water content of 0.6% and passed through a mesh of 60 mesh size, to 80 parts of the same powdery condiment as the control. The results of this test are set forth in Table 5. The sample according to this invention exhibited excellent stability without showing any discoloration even after 20 days of storage.

Table 5

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Classification	Stability as the lapse of storage days					
	2	4	8	12	15	20
Control	-	±	+	+	+	+
This Invention	-	-	-	-	-	-

Example 2

(1) Using as a control a powdery condiment (for pickles) prepared by admixing 44.5% of sodium L-glutamate, 10% of glycine, 2% of DL-alanine, 1.5% of succinic acid, 0.5% of citric acid (anhydrous) and 41.5% of fructose, each of the components having been passed through a mesh of 60 mesh size, a stability test under the same conditions as that described in Experimental Example 1 was conducted on

a composition prepared by adding 20 parts (44.2% relative to the total amount of sodium L-glutamate, glycine and DL-alanine) of purified and sterilized dry starch with a water content of 0.8% to the same condiment as the control.

- 5 The results are shown in Table 6. It is apparent that the composition of this invention exhibited excellent stability as compared with the control.

Table 6

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Classification	Stability as the lapse of storage days					
	2	5	10	14	17	20
Control	±	+	+	+	++	++
This invention	-	-	-	-	±	±

- (2) Twenty-five % of sodium glutamate, 2% of 5'-ribonucleotide sodium, 1% of 5'-guanylic acid, 1% of disodium succinate, 5% of glycine, 5% of DL-alanine, 10% of anhydrous glucose, 3% of sodium citrate and 48% (120% relative to the total amount of sodium glutamate, DL-alanine and glycine) of dry dextrin with a water content of 0.8%, each of them having been passed through a mesh of 60 mesh size, were evenly compounded at 15.6°C and at 55% R.H. to prepare a powdery condiment (for pickles).

- 100 g Portion each of thus prepared condiment was rapidly packaged gas-tight in a bag of laminate consisting of 12 μ polyester film, 13 μ polyethylene film, 9 μ aluminum laminate and 40 μ polyethylene film, and stored under atmospheric conditions at room temperature (15°C to 30°C) for 6 months, at the end of which time there was appreciated neither browning nor caking.

35

Example 3

(1) Using as a control a powdered juice (soft drink) prepared by admixing 40% of anhydrous glucose, 15% of glycine, 8% of DL-alanine, 34.094% of sucrose, 0.006% of vitamin B₁, 1.1% of sodium chloride, 1.1% of potassium chloride, 0.6% of disodium phosphate (anhydrous) and 0.1% of magnesium chloride, each of the components having been passed through a mesh of 60 mesh size, a stability test under the same conditions as that described in Experimental Example 1 was conducted on a composition prepared by adding 20 parts (108.7% based on the combined amount of glycine and DL-alanine) of β -cyclodextrin thermally dehydrated to a water content of 0.8% and passed through a mesh of 60 mesh size to 80 parts of the same powdered juice as the control. The results of the above test are shown in Table 7. It is apparent that whereas the control juice underwent browning and caking within a day after preparation, the composition according this invention exhibited excellent stability both in terms of discoloration and in terms of caking.

Table 7

Classification	Item of observation	Stability					
		1	4	7	10	16	20
Control	Browning	±	++	+++	+++		
	Caking*	±	+	+	++		
This invention	Browning	-	-	-	-	±	±
	Caking*	-	-	-	-	-	-

*: - denotes no caking, ± slightly caking,
+ through ++ various degrees of caking.

(2) Five % of sodium aspartate, 8% of glycine, 2% of DL-methionine, 1% of potassium chloride, 0.5% of magnesium sulfate (anhydrous), 31% of anhydrous glucose, 6% of purified stevioside, 3% of purified glycyrrhizin, 3.5% of disodium phosphate (anhydrous) and 40% (266% relative to the combined amount of sodium L-aspartate, DL-methionine and glycine) of dry dextrin with a water content of 0.8%, each of them having been passed through a mesh of 60 mesh size, were evenly admixed at 15.0°C and at 65% R.H. to obtain a powdery juice (soft drink).

100 g portion each of thus prepared powdery juice was rapidly packaged gas-tight in a bag of the laminate mentioned in Example 2.-(2), and stored under atmospheric conditions at room temperature (15°C to 30°C) for 6 months, at the end of which time there was appreciated neither browning nor caking.

Example 4

(1) Using as a control an electrolyte modifier for animal use prepared by admixing 47.39% of anhydrous glucose, 23.70% of glycine, 11.85% of sodium chloride, 7.11% of monopotassium phosphate, 4.74% of citric acid (anhydrous), 0.47% of magnesium sulfate (anhydrous) and 4.74% of sodium hydrogen carbonate, each of the components having been passed through a mesh of 60 mesh size, a stability test under the same conditions as that described in Experimental Example 1 was conducted on a composition by adding 100%, based on glycine, of dextrin [Pinedex] thermally dehydrated to a water content of 0.7% and passed through a mesh of 60 mesh size to the same electrolyte modifier as the control. As set forth in Table 8, the composition of this invention displayed excellent stability.

The control was dehydrated to a water content of 1% or less before gas-tight packaging.

Table 8

Classification	Stability as the lapse of storage days						
	4	7	10	12	16	20	30
Control	±	+	+	++	++	+++	+++
This invention	-	-	-	-	-	-	-

10

It is apparent that this invention provides a remarkable stabilization of the electrolyte modifier for animal use which consists of glucose, glycine and the electrolytes. The electrolyte modifier for animal use thus stabilized can be put to use in exactly the same manner as the conventional electrolyte modifier for animal use. For example, by dissolving it in water and administering the resulting solution to domestic animals, diarrhea in them can be prevented and cured.

(2) Thirty-six % of anhydrous glucose, 15.0% of glycine, 11.7% of sodium chloride, 2.3% of monopotassium phosphate, 8.6% of sodium citrate (anhydrous), 1.0% of magnesium sulfate (anhydrous), 8.4% of sodium hydrogen carbonate and 17.0% (113.3% relative to glycine) of dry dextrin with a water content of 0.8%, each of them having been passed through a mesh of 60 mesh size, were evenly compounded at 25°C and at 60% R.H. to prepare a powdery electrolyte modifier for animal use.

60 g Portion each of thus prepared electrolyte modifier was rapidly packaged gas-tight in a bag of the laminate mentioned in Example 2.-(2), and stored under atmospheric conditions at room temperature (15°C to 30°C) for 6 months, at the end of which time there was appreciated neither browning nor caking.

60 g Portion each of thus stabilized powdery electrolyte modifier is dissolved in 2l of water and orally

administered ad libitum to domestic animals, especially calves, suffered from diarrhea.

Example 5

5 Using as a control a powdered juice (soft drink)
 prepared by blending 55% of anhydrous glucose, 5% of sodium
 hydrogen carbonate, 3% of sodium L-aspartate, 5% of DL-
 alanine, 24.1% of sucrose, 1.1% of sodium chloride, 1.1% of
 10 potassium chloride, 0.6% of disodium phosphate (anhydrous),
 0.1% of magnesium chloride and 5% of citric acid (anhydrous),
 each of the components having been passed through a mesh
 of 60 mesh size, a stability test under the same conditions
 as that described in Experimental Example 1 was conducted
 on a composition prepared by adding 20 parts (312.5% based
 15 on the combined amount of sodium L-aspartate and DL-
 alanine) of β -cyclodextrin thermally dehydrated to a water
 content of 0.3% and passed through a mesh of 60 mesh size
 to the same juice as the control. The results are shown in
 Table 9. It is apparent that the composition according to
 20 this invention exhibited excellent stability without show-
 ing any discoloration or caking at all even after 20 days.

Table 9

25	Classification	<div>Storage days</div> <div>Item of observation</div>	Stability						
			2	4	8	11	15	20	30
	Control	Browning	-	<u>+</u>	+	++	++	+++	+++
		Caking	<u>+</u>	<u>+</u>	+	+	+	++	++
30	This invention	Browning	-	-	-	-	-	-	-
		Caking	-	-	-	-	-	-	-

What is claimed is:

1. A stabilized solid composition which contains one or more sugars selected from the group consisting of monosaccharide and disaccharide, one or more members of amino acid, and based on said amino acid member, at least 40% of one or more members of polysaccharide, said polysaccharide member having a water content of not more than 3%.
2. A composition of claim 1, wherein the polysaccharide member is composed of at least 4 monosaccharide units.
3. A composition of claim 2, wherein the monosaccharide unit is glucose unit.
4. A composition of claim 1, wherein the polysaccharide member is starch, pullulane, dextrin or/and cyclodextrin.
5. A composition of claim 1, wherein the polysaccharide member has a water content of not more than 1%.
6. A composition of claim 1, wherein the amount of the polysaccharide member is about 60% to 700% relative to the amino acid member.
7. A composition of claim 1, wherein the amount of the polysaccharide member is about 90% to 500% relative to the amino acid member.
8. A composition of claim 1, wherein the monosaccharide is tetrose, pentose or hexose.
9. A composition of claim 1, wherein the sugar member is glucose, fructose, xylose, galactose, sucrose, lactose or/and maltose.

10. A composition of claim 1, wherein the amino acid member is glycine, alanine, methionine, aspartic acid, glutamic acid or/and a sodium salt of them.
11. A composition of claim 1, which contains one or more members of electrolyte.
12. A composition of claim 11, the electrolyte member is acetate, carbonate or/and hydrogen carbonate.
13. A composition of claim 1, which is in powdery form.
14. A composition of claim 1, which contains glucose as the sugar member, glycine as the amino acid member, dextrin as the polysaccharide member and one or more members of electrolyte.
15. A composition of claim 13, wherein the electrolyte member is sodium chloride, monopotassium phosphate, sodium citrate, magnesium sulfate and sodium hydrogen carbonate.
16. A composition of any one of claims 1 to 15, which is in a gas-tight packaged unit usage form.
17. A method of stabilizing a solid composition containing one or more sugars selected from the group consisting of monosaccharide and disaccharide and one or more members of amino acid, which comprises incorporating in said solid composition at least 40%, based on said amino acid member, of one or more members of polysaccharide, said polysaccharide member having a water content of not more than 3%.



European Patent
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EUROPEAN SEARCH REPORT

0090356
Application number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 83102942.6
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
X	US - A - 2 790 719 (EARL B. GLICKMAN) * Claims; column 2, lines 49-73 *	1-4,8-13,15,17	A 23 L 1/227 A 23 L 1/228 A 23 K 1/18 C 08 L 5/00 C 08 L 3/02 C 08 L 5/16
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X	US - A - 2 774 673 (ROBERT H. YOUNG) * Column 2, lines 36-72; column 3, lines 23-33 *	1-4,8-11,15,17	C 07 H 3/C2 C 07 H 3/04 A 61 K 31/195 A 61 K 31/70 A 61 K 47/00
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The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 21-06-1983	Examiner WEIGERSTORFER
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	